Türkiye 16. Madencilik Kongresi / 76'* *Mining Congress of Turkey,* 1999, ISBN 975-395-310-0

# A NEW APPROACH FOR THE EXPLORATION AND EXPLOITATION OF MDLTI- VARI ABLE RESPONSE FUNCTIONS IN BATCH FLOTATION EXPERIMENTS

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ABSTRACT: The results of 74 rougher Flotation batch tests on a complex Pb-Zn-Ag sulfide ore are evaluated and analyzed with the help of powerful statistical tools available in electronic spreadsheets. The tests were designed by a combination of simultaneous and sequential experimental methods. Some of the statistically estimated interactions have plausible metallurgical interpretations. The main purpose of the study has been to determine whether multivariable experimental optimization methods can be used for increasing the efficiency of complex flotation operations and for gaining an enhanced understanding of the interactions involved. The results show promise in this regard but further batch and continuous studies must be performed, taking other factors into consideration, before application to me industrial operation can be realized.

## 1. INTRODUCTION

The ore used for the tests is made up of sphalerite, marmatite, galena and small quantities of tetrahedrite; the gangue consists of pyrite, marcasite, quartz, calcite and others.

Table 1 shows a typical reported Metallurgical Balance of the industrial operation.

Table 1. Typical Metallurgical Balance of die Industrial Operation

| | | Grades | | | Recoveries | | |
|---|---|---|---|---|---|---|---|
| Product | w% | Pb% | Zn% | Aqppm | Pb | Zn | Ag |
| Head (Calc) | 100.000 | 2.7 | 7.9 | B2 | 100.00% | 100.00% | 100.00% |
| Pb Concentrate | 3.969 | 54.1 | 4.1 | 952 | 79.53% | 2.06% | 46.08% |
| Zn Concentrate | 14.391 | 1.06 | 49.0 | 90 | 5.65% | 89.26% | 15.79% |
| Tailing | 81.640 | 0.49 | 0.84 | 38 | 14.82% | 8.68% | 37.83% |

The low recovery of Ag in the Pb concentrate (46.08%) points to the need of optimizing the lead circuit. In this process the rougher flotation circuit is critical: its objective İs to float Pb and Ag and to depress Zn, in the maximum measure possible consistent with the global economic objective of the operation. The latter depends on the relative prices of the valuable metals contained. Since the batch tests are made under different conditions than a continuous operation (no closed circuits in grinding or flotation) we did not expect that their efficiency would approach that of the industrial operation. The objective here is to maximize *relative* efficiency within the context of the batch tests and to identify the main interactions involved which may guide the researcher towards obtaining better answers for the industrial operation.

## 2. EXPERIMENTAL SETUP

The batch charge consisted of 500 grams of ore previously crushed to less than 10 mesh. Grinding time was variable. The average particle size approx. 60% minus 200 mesh. Ball charge was constant in the tests. The head material was not perfectly homogenized. The reagents added at the grinding stage, together with 500cc of water, were CaO (modifier), A-3418 (promoter), $CNNa+ZnS0_4$ (depressants, in fixed 1:3 proportion). M1BC (fromer) and Z-11 (collector) were added to the Flotation cell and given 3 minutes of conditioning. The other controllable variable is flotation time that varied among tests according to the experimental design. Z-11, A3418 and MIBC were added at 1% $H_20$ dilution, and NaCN and $ZnS0_4$ at 5% dilution.

## 3. OBJECTIVE EFFICIENCY CRITERION

Given the proposed objective of the first stage of this flotation process, an efficiency index for our batch tests can be defined as follows:

$$\exists [ \forall R_{Pb} + (1-\forall)R_{Ag} ] + (1-\exists)(1 - R_{Zn}) \qquad (1)$$

where Rpb = Recovery of Pb İn the Pb concentrate
RAg = Recovery of Agin the Pb concentrate
Rzn = Recovery of Zn in the Pb concentrate
V = weight factor reflecting the economic importance of Pb with respect to Pb+Ag in the Pb concentrate. The average used was 0.52 (depends on head grades and relative prices).
3 = weight factor reflecting the economic importance of Pb+Ag with respect to Pb+Ag+Zn in the global operation. The average used for 3 was about 0.36 showing that Zn is the main economic component of the head material.

A valid criticism of this efficiency function would be that high recoveries of Fe are not penalized. Other improvements to the efficiency index could also be considered, such as including variable costs of reagents, incremental capital costs when residence times increase, etc. However, this simplified efficiency index will serve our purpose for illustrating the potential of the multivariable methods for searching improved solutions for the separation problem.

## 4. PRELIMINARY TWO-VARIABLE DESIGNS

Table 2 shows a preliminary Hexagonal Design varying only A3418 and Grinding Time.

Table 2. Hexagonal Design

| Test* | A3418 | TJarind | %S.E. | Predicted |
|---|---|---|---|---|
| HOI | 2 | 10.5 | 83.86 | 83.465 |
| K018 | 1.9 | 10 | 82.58 | 82.907 |
| H03 | 1.6 | 10 | 82.72 | 82.394 |
| H04 | 1.5 | 10.5 | 82.5 | 82.893 |
| H05 | 1.6 | 11 | 82.78 | 82.453 |
| H06 | 1.9 | 11 | 82.3 | 82.628 |
| H07 | 1.8 | 10.5 | 82.61 | 62.998 |
| H016 | 1.8 | 10.5 | 82.57 | 82.998 |
| H017 | 1.8 | 10.5 | 83.61 | 82.998 |

Table 3 shows that the results of this Hexagonal Design have a very poor statistical significance.

Table 3. Quadratic model for Hexagonal Design

| Rearesslon Statistics | | | | |
|---|---|---|---|---|
| R Square | 39.06% | | | |
| Adjusted $R^2$ | -62.52% | | | |
| Std Error | 0.6697 | | | |
| Observations | 9 | | | |
| **Analysis of Variance** | | | | |
| | ***df*** | ***SumSq*** | ***MeanSq*** | ***F*** |
| Regression | 5 | 0.866 | 0.173 | 0.384 |
| Residual | 3 | 1.330 | 0.443 | |
| Total | 8 | 2.197 | | |
| | ***Coeff.*** | ***Std Err*** | ***f value*** | ***P-value*** |
| Intercept | -113.483 | 221.01 | -0.51 | 62.2% |
| xi | -0.957 | 58.97 | -0.02 | 98.7% |
| *2 | 37.491 | 38.91 | 0.96 | 36.3% |
| $x,^2$ | 3.979 | 10.26 | 0.39 | 70.8% |
| x/ | -1.697 | 1.81 | -0.93 | 37.7% |
| x,x? | -1.126 | 4.46 | -0.25 | 80.7% |

!n the following Octogonal design the range of variables is twice that of the previous design.

Table 4. Octogonal Design

| Test# | A3418 | T Grind | %S.E. | Predicted |
|---|---|---|---|---|
| H08 | 2.2 | 10.50 | 83.13 | 83.074 |
| H09 | 2.1 | 9.63 | 83.25 | 83.279 |
| HO10 | 1.8 | 9.50 | 82.68 | 62.861 |
| H011 | 1.4 | 9.63 | 82.05 | 82.047 |
| H012 | 1.3 | 10.50 | 82.26 | 82.260 |
| H013 | 1.4 | 11.37 | 82.25 | 82.264 |
| H014 | 1.8 | 11.50 | 82.37 | 82.320 |
| H015 | 2.1 | 11.37 | 82.24 | 82.297 |
| H07 | 1.8 | 10.50 | 82.81 | 83.008 |
| H016 | 1.8 | 10.50 | 82.57 | 83.008 |
| H017 | 1.8 | 10.50 | 83.61 | 83.008 |

Table 5. Quadratic model for Octogonal Design

| Rearesslon Statistics | | | | |
|---|---|---|---|---|
| R Square | 75.64% | | | |
| Adjusted $R^2$ | 51.28% | | | |
| Std Error | 0.349 | | | |
| Observations | 11 | | | |
| **Analysis of Variance** | | | | |
| | ***df*** | ***SumSq*** | ***MeanSq*** | F |
| Regression | 5 | 1.8965 | 0.3793 | 3.105 |
| Residual | 5 | 0.6107 | 0.1221 | |
| Total | 10 | 2.5073 | | |
| | ***Coeff.*** | ***Std Err*** | ***t value*** | ***P-value*** |
| Intercept | 14.9164 | 30.1846 | 0.49 | 63.19% |
| $x_t$ | 16.4751 | 7.6738 | 2.15 | 5.74% |
| % | 10.2866 | 5.3519 | 1.92 | 8.35% |
| X1 | -1.4822 | 1.3611 | -1.09 | 30.17% |
| x/ | -0.4180 | 0.2501 | -1.67 | 12.56% |
| XiXj | -0.9889 | 0.5742 | -1.72 | 11.57% |

The statistical significance in this case is better than that of the Hexagonal design but not yet quite satisfactory (the significance of the F statistic is about 12%). In terms of metallurgical significance we do not see much variation in the predicted values of the Separation Efficiency (1.2% range). Figure 1 shows the surface graph of the Octogonal design.

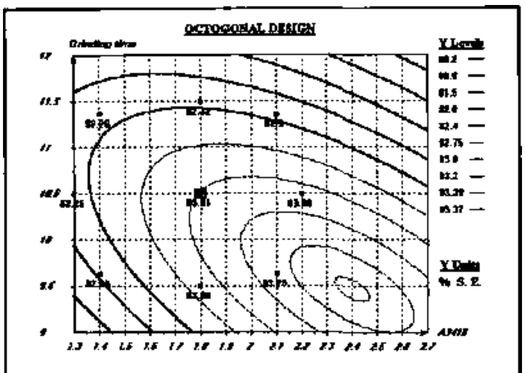


Figure 1: Response Surface from Octogonal Design.

Combining the data from both designs we can use an expanded model which takes into account effects and interactions of non-controllable variables such as head grades (x3, X4, X5). After eliminating some statistically unsignificant parameters we obtain the following model results and surface graph:

Table 6. Quadratic model for Combined Design

| *Rearessfon Statistics* | | | | |
|---|---|---|---|---|
| R Square | | 83.41% | | |
| Adjusted $R^2$ | | 62.67% | | |
| Std Error | | 0.301 | | |
| Observations | | 19 | | |
| ***Analysis of Variance*** | | | | |
| | ***dt*** | ***SumSq*** | ***MeanSq*** | ***F*** |
| Regression | 10 | 3.633 | 0.363 | 4.02 |
| Residual | 8 | 0.723 | 0.090 | |
| Total | 18 | 4.356 | | |
| | ***Coeff.*** | ***Std Err*** | ***t value*** | ***P-value*** |
| Intercept | 82.842 | 0.128 | 647.35 | 0.00% |
| Xi | 0.214 | 0.341 | 0.63 | 53.83% |
| % | -0.053 | 0.188 | -0.28 | 77.97% |
| $<_2$ | -0.870 | 1.133 | -0.77 | 45.28% |
| $xa^2$ | -0.312 | 0.260 | -1.20 | 24.51% |
| X3 | 3.071 | 1.565 | 1.96 | 6.55% |
| X4 | -5.920 | 1.625 | -3.64 | 0.19% |
| Xs | -0.254 | 0.595 | -0.43 | 67.46% |
| X1X6 | -2.688 | 2.766 | -0.97 | 34.40% |
| X7X3 | -6.237 | 2.895 | -2.15 | 4.50% |
| X2X4 | 9.057 | 4.059 | 2.23 | 3.86% |

The predicted responses for the "Combined" response function are calculated by holding the uncontrolled variables (the head grades: $x_($=Pb, x:=Ag, and X3=Zn) constant while evaluating the Y values.

This model has a better statistical performance than the previous ones: the "F' statistic is significant at the 3% level. However, the predicted maxima both in the Octogonal and the Combined cases lie on an somewhat "flat" surface, i.e. the predicted maxima are not significantly greater than many of the original design points.

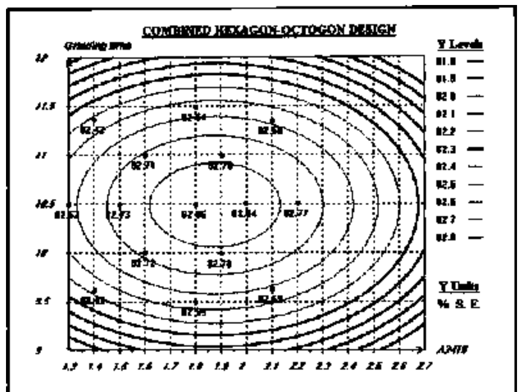


Figure 2: Response Surface from Combined Design.

## 5. INITIAL SEVEN-VARIABLE DESIGN

Table 7 presents the values of the controllable variables and the results of the initial design in terms of five responses: Separation Efficiency (S.E.), and the partial metallurgical recoveries of Pb, Ag, Zn and Fe in the Pb concentrate. "CN+ZS" refers to the compound variable NaCN+ZnS04 whose components were added in fixed proportions (1:3).

In the Response section we can observe that the S.E. has a relative variation of 1.24% (standard deviation divided by the average). In comparison the individual recoveries vary as follows: Pb-1.85%, Ag-6.1%, Zn-32.5%, and Fe-39.2%. The relative stability of S.E. in comparison to the recoveries is due mainly to the fact that greater recoveries of Pb and Ag are in most cases offset by greater Zn recovery. In other words, some of the factors contributing to a greater recovery of Pb and Ag also contribute to a greater recovery of Zn. Also, it can be shown that a significant part of the response variance is caused by the variations in head grades among tests since heads were not homogenized.

Table 7. Results of Initial 7 variable Double Simplex Design (plus two replicates)

| | Controllable Variables | | | | | | | Ohşetved ResDonses f% values | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Test# | Z11 | A341Ş | MIBC | CN+ZS | CaO | TFlot | Tarind | S.E. | R-Pb | H-AQ | R-Zn | R-Fe |
| SI-1 | 1 | 1.6 | 1.3 | 4.4 | 1.83 | 2.36 | 9.5 | 79.91 | 72.98 | 45.52 | 7.76 | 8.27 |
| SI-2 | 2 | 1.6 | 1.3 | 4.4 | 1.83 | 2.36 | 9.5 | 80.63 | 74.38 | 51.97 | 9.57 | 14.83 |
| SI-3 | 1.5 | 2.8 | 1.3 | 4.4 | 1.83 | 2.36 | 9.5 | 81.64 | 71.87 | 47.90 | 6.52 | 10.85 |
| SI-4 | 1.5 | 2 | 2 | 4.4 | 1.83 | 2.36 | 9.5 | 81.72 | 72.47 | 45.87 | 5.49 | 6.07 |
| SI-5 | 1.5 | 2 | 1.5 | 4.4 | 1.83 | 2.36 | 12.0 | 81.81 | 72.87 | 48.04 | 6.40 | 9.14 |
| SI-6 | 1.5 | 2 | 1.5 | 8 | 1.83 | 2.36 | 10.0 | 82.26 | 71.83 | 47.02 | 5.51 | 8.61 |
| SI-7 | 1.5 | 2 | 1.5 | 5 | 3 | 2.36 | 10.0 | 83.45 | 74.75 | 45.60 | 4.20 | 5.31 |
| SI-8 | 1.5 | 2 | 1.5 | 5 | 2 | 3.50 | 10.0 | 81.59 | 72.88 | 45.60 | 6.24 | 7.64 |
| SI-9 | 2 | 2.4 | 1.7 | 5.6 | 2.17 | 2.64 | 10.5 | 81.87 | 72.29 | 46.82 | 5.87 | 7.65 |
| SI-10 | 1 | 2.4 | 1.7 | 5.6 | 2.17 | 2.64 | 10.5 | 82.42 | 73.33 | 47.65 | 5.50 | 7.63 |
| SI-11 | 1.5 | 1.2 | 1.7 | 5.6 | 2.17 | 2.64 | 10.5 | 81.83 | 74.94 | 50.69 | 7.52 | 8.15 |
| SI-12# | 1.5 | 2 | 1 | 5.6 | 2.17 | 2.64 | 10.5 | 82.09 | 74.85 | 45.55 | 5.51 | 6.39 |
| SI-13 | 1.5 | 2 | 1.5 | 5.6 | 2.17 | 2.64 | 8.0 | 80.64 | 74.36 | 46.37 | 7.70 | 9.26 |
| SI-14 | 1.5 | 2 | 1.5 | 2 | 2.17 | 2.64 | 10.0 | 79.05 | 75.04 | 56.21 | 13.89 | 15.24 |
| SI-15 | 1.5 | 2 | 1.5 | 5 | 1 | 2.64 | 10.0 | 82.25 | 71.66 | 51.95 | 6.94 | 20.33 |
| SI-16 | 1.5 | 2 | 1.5 | 5 | 2 | 1.50 | 10.0 | 81.70 | 69.84 | 44.69 | 4.71 | 9.35 |
| SI-17 | 1.5 | 2 | 1.5 | 5 | 2 | 2.50 | 10.0 | 82.95 | 73.41 | 50.51 | 5.81 | 8.35 |
| si-1 en | 1.5 | 2.1 | 1.5 | 4 | 2 | 3.00 | 9.634 | 81.59 | 73.45 | 47.97 | 6.86 | 9.17 |
| SI-19 # | 1.5 | 2 | 1 | 5.6 | 2.17 | 2.64 | 10.5 | 83.16 | 71.98 | 45.27 | 3.96 | 5.16 |

# These tests are repeated (") Repetition of Test HO-9 from the Octogonal design

The above Table clearly illustrates the effects of adding low quantities of two important variables: in Test SI-14 the low dosage of "CN+ZS" causes a great increase in the recoveries of Zn and Fe. On the other hand the low CaO dosage İn Test SI-15 causes an even sharper increase in the recovery of Fe, although in this case Zn recovery is only slightly higher than the average. This points to a differentiated role of these two variables in the depression of Zn and Fe, which will be exploited as we progress in the sequence of experiments.

## 6. SECOND SEVEN-VARIABLE DESIGN

The second group of tests (labelled A1) is derived from the results of the first group (labelled SI) according to a pattern analogous to SSDEVOP or Simplex Search (Mular, 1976), which *aie sequential,* as opposed to *simultaneous,* methods. The difference here İs that multiple points are developed at each stage instead of only one in order to save time. This is because it takes almost as much time to analyze one sample as 20 samples. The results of the second group of tests are shown in Table 8.

Table 8. Results of Second 7 variable Design (plus two replicates)

| | Controllable Variables | | | | | | | Observed Resoonses (% values* | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Test# | Z11 | <\341ß | MIBC | CN+ZS | CaO | TFlot | Tarind | S.E. | R-Pb | R-Aq | R-Zn | R-Fe |
| M-1 | 1.3 | 1.8 | 1.9 | 6.8 | 3.07 | 2.41 | 9.4 | 80.90 | 72.02 | 43.87 | 5.30 | 5.26 |
| M-2 | 0.8 | 1.5 | 1.6 | 6 | 1.55 | 2.45 | 9 | 80.61 | 73.61 | 52.32 | 9.04 | 13.23 |
| M-3 | 1.5 | 2 | 1.8 | 6.4 | 1.81 | 1.62 | 9.7 | 82.11 | 69.97 | 46.18 | 4.72 | 7.18 |
| M-4 | 1.5 | 2 | 1.8 | 6.4 | 1.84 | 2.85 | 9.8 | 81.84 | 76.05 | 54.41 | 9.01 | 12.06 |
| M-5 | 2 | 2.4 | . 1.9 | 6.8 | 2.02 | 2.96 | 10.3 | 82.64 | 74.45 | 49.18 | 6.11 | 9.00 |
| M-6 | 1.4 | 1.9 | 1.7 | 6 | 1.83 | 3.76 | 9.7 | 82.89 | 77.23 | 52.83 | 7.47 | 9.36 |
| M-7# | 1.4 | 1.9 | 1.7 | 5.6 | 0.85 | 2.69 | 9.7 | 82.92 | 72.64 | 55.40 | 7.39 | 21.06 |
| M-8 | 1.4 | 1.9 | 1.7 | 6.4 | 2.2 | 2.67 | 7.8 | 82.99 | 73.13 | 50.48 | 6.15 | 7.11 |
| M-9 | 1.4 | 1.1 | 1.8 | 6 | 2.17 | 2.64 | 10.6 | 82.60 | 73.18 | 49.71 | 5.96 | 7.36 |
| M-10 | 1.4 | 2.1 | 2.1 | 4.8 | 1.81 | 2.34 | 9.5 | 81.14 | 73.70 | 50.92 | 8.12 | 12.03 |
| M-11 | 0.9 | 2.5 | 1.7 | 6 | 2.17 | 2.68 | 10.6 | 81.78 | 73.32 | 48.42 | 6.46 | 7.76 |
| M-12 | 1.5 | 2 | 1.5 | 8.4 | 1.81 | 2.34 | 10 | 81.33 | 73.02 | 49.23 | 6.90 | 13.00 |
| M-13 | 1.4 | 1.9 | 1.8 | 6.4 | 1.93 | 2.66 | 9.7 | 81.59 | 74.37 | 51.94 | 8.36 | 10.38 |
| M-U(*) | 1 | 2.4 | 1.7 | 5.6 | 2.17 | 2.64 | 10.5 | 82.14 | 75.24 | 50.23 | 7.26 | 9.03 |
| M-15# | 1.4 | 1.9 | 1.7 | 5.6 | 0.85 | 2.69 | 9.7 | 83.11 | 72.42 | 54.74 | 6.18 | 22.60 |

#These tests are repeated OF Repetition of TestSMOftom the Initial Double Simplexclesion

Tests Al-7 and Al-15 show that low CaO with sufficient "CN+ZS" addition increase Fe recovery while Zn recovery remains at an acceptable level. Also Ag recovery seems to be somewhat correlated with Fe recovery (76% simple correlation).

At this stage we used the accumulated data of series SI and Al to derive the quadratic regression shown in Table 9, after selecting the more significant variables by a process of trial-and-error.

Table 9. Regression model 1 for 7-Variable Design

| *Rearession Statistics* | | | | |
|---|---|---|---|---|
| Ft Square | | 93.06% | | |
| adjusted $R^2$ | | 84.73% | | |
| Standard Error | | 0.377 | | |
| Observations | | 34 | | |
| ***Analysis of Variance*** | | | | |
| | ***Of*** | ***SumSq*** | ***MeanSq*** | ***F*** |
| Regression | 18 | 28.5502 | 1.5661 | 11.17 |
| Residual | 15 | 2.1291 | 0-1419 | |
| Total | 33 | 30.6793 | | |
| | ***Coeff.*** | ***StdErr*** | ***t value*** | ***P-value*** |
| Intercept | 395.080 | 96.9319 | 4.08 | 0-03% |
| Z-11 | 1.1259 | 1.7689 | 0.64 | 52.88% |
| A3418 | 1.2397 | 1.0042 | 1.23 | 22.57% |
| MIBC | 5.7938 | 2.6579 | 2.18 | 3-657= |
| CN | 5.3864 | 1.2203 | 4.41 | 0-01% |
| CaO | -46.8976 | 16.2934 | -2.88 | 0.70% |
| T Flot | 0.8278 | 0.5303 | 1.56 | 12.80% |
| T Grind | 0.2351 | 0.0971 | 2.42 | 2.11% |
| Pb | 1.8510 | 1.4921 | 1.24 | 22.35% |
| Ag | •7.4554 | 1.2826 | -5.81 | 0.00% |
| Zn | -77.3133 | 23.9826 | -3.22 | 0-28% |
| Z1TA3418 | -0.8997 | 0.7165 | -1.26 | 21.80% |
| Z11/T Flot | 2.5036 | 1.9325 | 1.30 | 20.41% |
| MIBC'CN | -1.0057 | 0.5100 | -1.97 | 5.70% |
| CN* | -0.1731 | 0.0343 | -5.04 | 0.00% |
| CN'CaO | -0.7706 | 0.3668 | -2.10 | 4.34% |
| $CaO^2$ | 2.2719 | 0.7448 | 3.05 | 0.45% |
| CaO'Zn | 5.6799 | 1.9700 | 2.88 | 0.69% |
| $Zn^2$ | 4.5225 | 1.4829 | 3.05 | 0.45% |

The high significance of the positive coefficient for $CaO^2$ (t=3.05, 0.45% p-value), shows that within the experimental range our estimated response function is not strongly unimodal, thus making any optimization procedure the more difficult.

The F statistic for this model is significant at less than 0.01% level. However, this is no guarantee that predictions will be accurate, since there may be a high degree of lack of fit. Nevertheless we ventured to use a numerical optimization procedure to maximize an objective function, subject to the constraints that the controllable variables do not exceed the minimum or maximum values taken by the seven variables in stages SI and AI. The objective function is the scalar product of the regression coefficients' vector and the (unknown) vector of values taken by the seven controllable variables and the composite variables derived thereof. The respective upper and lower limits of the controllable variables were:

| | Z-11 | A3418 | MIBC | CN+ZS | CaO | T„Flot | TJ3rind |
|---|---|---|---|---|---|---|---|
| Max' | 2 | 2.8 | 2.1 | 8.4 | 3.07 | 3.76 | 12 |
| Min | 0.8 | 1.1 | 1 | 2 | 0.85 | 1.50 | 7.8 |

If we attempt an unconstrained maximization of this function we will obtain an unbounded solution (the predicted S.E. goes to infinity). The constrained solution to this problem was the following:

| Z-11 | A3418 | MIBC | CN+ZS | CaO | T_Flot | T_Grind |
|---|---|---|---|---|---|---|
| 2 | 1.1 | 1 | 8.4 | 0.85 | 3.76 | 12 |

## 7. THIRD SEVEN-VARIABLE DESIGN AND FINAL VERIFICATION TESTS

Table 10 shows the results of the next group of tests, designed according to our special sequential method and supplemented by test A2-15 (programmed by the constrained method of the previous section) plus a couple more tests designed for filling in some gaps. We were fortunate that the best result so far was obtained in test A2-I5. Clearly, using more CN+ZS together with less CaO we obtain smaller Zn and greater Ag recoveries, thus increasing S.E.

The next step involved replicating tests A2-15, SI-7, SI-I7 and A1-8. An approximate confidence interval (Draper & Smith, 1966) for the expected responses of each of these tests can be calculated using the following formula:

$$\psi_0 = \int_0 \pm s_r t_{\alpha,dl} \, SQRT(x_0(X'X)^{-1}x_0') \qquad (2)$$

where

$\psi_0$ = confidence interval.

$\int_0$ = predicted value of response.

$s_r$ = Standard error of Y estimate in regression.

$t_{\alpha,dl}$ = value in Student's "t" table at rt% level of significance with dl degrees of liberty.

Xo= vector of values of independent variables for which prediction is made.

X = matrix of values of observed independent variables used in estimating the coefficients for the regression model (includes column of ones).

Table 10. Results of Third 7 variable Design (plus one predicted optimum test and three more "free" tests)

| | Controllable-Variables | | | | | | | Observed Responses (% valuesl | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Tesr# | Zl 1 | A3418 | M1BC | CN+ZS | CaO | TFlot | Tgrind | S.E. | R-Pb | R-Ae | R-Zn | R-Fe |
| A2-1 | 1.5 | 2 | 1.5 | 7.6 | 2.39 | 1.11 | 9.900 | **76.69** | 62.46 | 30.20 | 2.93 | 3.35 |
| A2-2 | 1.5 | 2.8 | 1.5 | 7.2 | 1.96 | 2.66 | 9.000 | **8O.S0** | 73.97 | 48.41 | 8.47 | 13.20 |
| A2-3 | 2 | 1.1 | 1.6 | 7.2 | 1.99 | 2.62 | 9.200 | **79.57** | 74.43 | 46.57 | 8.21 | 12.92 |
| A2-4 | 1.2 | 2.6 | 1.6 | 7.2 | 2.01 | 2.67 | 9.500 | **78.85** | ' 74.42 | 46.13 | 8.72 | 12.90 |
| A2-5 | 1.3 | 1.6 | 1.5 | 6.4 | 2.36 | 1.45 | 10.300 | **81.57** | 71.45 | 47.38 | 6.15 | 7.76 |
| A2-6 | 1.4 | 1.8 | 1.7 | 6.4 | 1.95 | 2.68 | 12.300 . | **81.07** | 74.17 | 52.57 | 9.07 | 11.61 |
| A2-7 | 1.8 | 1.3 | 1.7 | 7.2 | 2 | 2.71 | 9.200 | **81.41** | 74.89 | 52:45 | 8.72 | 12.60 |
| A2-8 | 0.7 | 1.4 | 1.5 | 6 | 2.17 | 2.38 | 9.500 | **82.41** | 73.91 | 49,54 | 6.68 | 6.93 |
| A2-9 | 1.3 | 1.9 | 1.6 | 6.4 | 2.36 | 3.76 | 10.100 | **82.16** | 76.66 | ,63.48 | 8.76 | 11.27 |
| A2-10 | 1.3 | 1.9 | 1.9 | 7.6 | 2.13 | 1.73 | 9.800 | **81.54** | 71.27 | »4:7.72 | 6.13 | 9.98 |
| A2-11 | 2 | 2.4 | 1.8 | 6.4 | 2.58 | 2.90 | 10.800 | **8133** | 71.77 | 46.31 | 6.47 | 8.33 |
| A2-12 | 1.4 | 1.7 | 1.3 | 7.6 | 2.25 | 2.99 | 10.200 | **81.86** | 75.78 | 49.29 | 7.47 | 8.62 |
| A2-13 | 1.4 | 1.9 | 1.6 | 6.8 | 2.18 | 2.56 | 10.000 | **81.96** | 74.42 | 48.89 | 6.85 | 9.18 |
| A2-14 | 1 | 2.2 | 1.5 | 5.6 | 2.1 | 2.50 | 10.500 | **81.74** | 74.29 | 51.72 | 7.85 | 9.97 |
| **A2-15** | 2 | **1.1** | 1 | **S.4** | **0.85** | **3.76** | **12.000** | **84.09** | **74.82** | **57.25** | **6.10** | **23.99** |
| A2-16 | 2 | 1.1 | 1.4 | 6.8 | 3.07 | 3.76 | 12.000 | **81.83** | 73.88 | 48.40 | 6.73 | 8.11 |
| A2-17 | 1.3 | 1.6 | 1.5 | 6.4 | 2.36 | 2.45 | 10.300 | **81.93** | 73.53 | 51.81 | 7.56 | 12.56 |

In order to calculate the predicted responses and confidence intervals we use the test data in series SI, Al and A2. Table 11 shows the regression model obtained by selecting the more significant variables:

Table 11. Regression model II for 7-Variable Design

| ***Regression Statistics*** | |
|---|---|
| R Square | 96.95% |
| Adjusted $R^2$ | 94.55% |
| Standard Error | 0.291 |
| Observations | 51 |

***Analysis of Valance***

| | *ttf* | *Sum Sq* | *Mean $q* | *F* |
|---|---|---|---|---|
| Regression | 22 | 75.4726 | 3.4306 | 40.46 |
| Residual | 28 | 2.3739 | 0.0848 | |
| Total | 50 | 77.8464 | | |

This model is the best so far from a statistical point of view. It has the lowest standard error for the Y estimate (0.291), the highest $R^2$ (96.95%) and the most significant F (40.5).

Here we note the presence of new statistically significant variables, notably a third order interaction and the variable "Weight". We were forced to include the latter "uncontrolled" variable in the model since due to an inadvertent error in calibrating the balance, the average weight of the batch charges in series A2 turned out to be 514 g instead of an average of 491 g in the previous tests.

Table 11. (Continued)

| | *Coeff.* | *StdErr* | *t value* | *P-value* |
|---|---|---|---|---|
| Intercept | 306.523 | 60.453 | 5.07 | 0.00% |
| 2-11 | 4.762 | 1.318 | 3.61 | 0.07% |
| A3416 | -0.300 | 0.121 | -2.48 | 1.64% |
| MIBC | 29.389 | 8.137 | 3.61 | 0.07% |
| CN | 3.238 | 1.304 | 2.48 | 1.64% |
| CaO | -27.851 | 7.210 | -3.86 | 0.03% |
| T Flot | 1.200 | 0.714 | 1.68 | 9.90% |
| T Mol | 0.154 | 0.057 | 2.70 | 0.95% |
| Pb | 16.956 | 4.834 | 3.51 | 0.10% |
| Ag | -17.558 | 4.510 | -3.89 | 0.03% |
| Zn | -59.155 | 15.461 | -3.83 | 0.04% |
| Weight | -0.030 | 0.005 | -6.01 | 0.00% |
| Z11/T Flot | -3.291 | 0.804 | -4.09 | 0.02% |
| MIBC*CaO | -2.702 | 0.793 | -3.41 | 0.13% |
| MIBC'Zn | -3.165 | 1.038 | -3.05 | 0.37% |
| $(CN+ZS)^2$ | -0.087 | 0.032 | -2.67 | 1.02% |
| (CN+ZS)*Pb | -2.099 | 0.786 | -2.67 | 1.02% |
| (CN+ZS)*Ag | 1.488 | 0.641 | 2.32 | 2.43% |
| $CaO^2$ | 1.167 | 0.291 | 4.01 | 0.02% |
| CaO'Tflot | -0.853 | 0.357 | -2.39 | 2.08% |
| CaO*Zn | 4.289 | 0.965 | 4.44 | 0.00% |
| CaO*Z1TAg | -0.784 | 0.282 | -2.78 | 0.76% |
| $Zn^2$ | 3.785 | 1.034 | 3.66 | 0.06% |

An example of an interesting interaction in this model is that of (CN+ZS)*Pb (i.e. the interaction between the dosage of Sodium Cyanide plus Zinc Sulfate and the grade of Lead in the Head material). The verbal explanation for this significantly negative parameter (t=-2.67, 1% level of significance) is that when the head grade of Pb increases, a *reduction* in the dosage of CN+ZS will cause an *increase* in S.E., and vice-versa. Figure 3 shows an order of magnitude of the predicted effects of this interaction: at 2.5% head grade of Pb the optimum dosage of CN+ZS is 7.5cc, and at 2.3% the optimum dosage increases to 9.7cc, all else remaining equal.

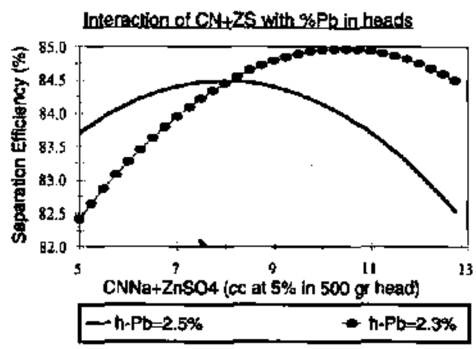


Figure 3: Effect of h-Pb on optimum dosage of CN.

Table 12 shows the predicted S.E. for the final tests with their 95% and 99% confidence intervals (Equation 2) and Table 13 shows the values of their controllable variables and observed responses. The complete raw data for series SI, AI, A2 and the verification tests is given in the Appendix.

Table 12. Predicted S.E.'s and Confidence Intervals

| rest# | % S.E. | 95% LL | 95%UL | 99% LL | 99%UL |
|---|---|---|---|---|---|
| R-1 (rSI-7) | 82.45 | 81.56 | 83.35 | 61.24 | 83.66 |
| R-2(rSI-17) | 80.99 | 80.71 | 81.27 | 80.61 | 81.37 |
| R-3(rA1-8) | 81.38 | 81.10 | 81.66 | 81.00 | 81.76 |
| R-4(rA2-15) | 84.61 | 83.71 | 85.51 | 83.39 | 85.82 |

It must be noted that all predictions in Table 12 have been calculated for *constant* head grades. On the other hand, all responses shown in Table 13 are given as observed, that is, their values are affected by the variable head grades (remember that our input material was not perfectly homogenized). This partially explains the fact that a couple of observed responses in Table 13 lie outside the confidence intervals of Table 12.

Table 13. Results of Verification Tests

| | Controllable Variables | | | | | | | Observed Resrjonses (% values) | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Test# | Z11 | A3418 | MIBC | CN+ZS | CaO | Tflot | Tarind | | R-Pb | R-Aa | R-Zn | R-Fe |
| R-1 | 1.5 | 2 | 1.5 | 5 | 3 | 2.36 | 10.0 | 82.78 | 68.10 | 43.54 | 4.04 | 5.10 |
| R-2 | 1.5 | 2 | 1.5 | 5 | 2 | 2.50 | 10.0 | 83.13 | 71.96 | 52.04 | 5.17 | 17.17 |
| R-3 | 1.4 | 1.9 | 1.7 | 6.4 | 2.2 | 2.67 | 7.8 | 82.59 | 73.99 | 51.55 | 6.77 | 7.92 |
| R-4 | 2 | 1.1 | 1 | 8.4 | 0.85 | 3.76 | 12.0 | 84.11 | 75.17 | 58.01 | 6.10 | 23.00 |

## 8. CONCLUSIONS

According to previous simulations with 7 variable unimodal test functions the sequential search method should give improved results starting from the second group of tests. In our investigation the method did not work as expected, so we used a complementary method by optimizing a prediction model.

There are two main difficulties for finding the optimum combination of controllable variables for this problem. First, some of the factors leading to an increase in the recovery of Pb and Ag also lead to an increase in the recovery of Zn; and second, mere is clear evidence that the response function is not strongly unimodal, and probably is multimodal.

The number of tests made is too small for an adequate characterization of the response function. Ideally the ratio of observations to regression coefficients should be at least 2:1 and preferably around 3:1. Since there are at least 70 potentially significant first and second order effects and interactions İt would be necessary to perform at least 150 tests for adequately estimating the desired parameters. The ideal mathematical situation is to be able to arrive at an "unconstrained optimization" and thus avoid bounding the controllable variables by arbitrary numbers, as we were forced to do with model I. Otherwise it will always be possible to improve the solution by doing more tests.

Further testing would eventually permit us to '"exploit" the response function more thoroughly, that is, to obtain even better results for the "separation efficiency" of the process. The high cost of performing such a large number of batch Flotation tests could be easily offset by the benefits of such an investigation. Let us assume that the insights obtained thereof lead to a 1% increase in Economic Recovery (Weiss, 1962). The ore we are working with has an "ideal" recovery value of approximately $40/ton. In this case the incremental revenue per ton of Head would be $0.40 which multiplied by 6,000 TPD would amount to $2,400 per day. Performing 150 Flotation tests should cost no more than $15,000 that would be recovered in less than a week assuming the above parameters. However, synchronization between Mine and Plant is crucial, otherwise by the time we finish optimizing one mineral its source might be already depleted- This leads us to the following conclusion.

The effectiveness of experimental optimization in Flotation can be dramatically enhanced with a state-of-the-art X-Ray Fluorescence (XRF) spectrometer, by reducing assay costs and speeding up the whole process of experimentation. There would be extra benefits if the offline X-Ray equipment is located near the Plant. It could serve not only for routine analyses required for Plant control and non-routine analyses {experimental work and circuit evaluations), but also for Mine production control and Geologic exploration purposes, thus reducing the unit cost of chemical analyses. Modem XRF systems are easily calibrated for accurate determinations of Pb, Zn, Ag and also for Cu, Fe, As, Sb, Bi, S, and other elements which are usually important for polymetallic mines.


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## APPENDIX

Raw data for tests of series SI, A1. A2 and R.

| [fiât | Wt. of Head | Wt. Of Cone. | Concentrate Pb (%) | Concentrate Ag (ppm> | Concentrate Zn (%) | Tailing Pb (%) | Tailing Ag <D0m> | Tailing Zn (%) |
|---|---|---|---|---|---|---|---|---|
| Si-1 | 493.5 | 38.3 | 23.1 | 421 | 7.13 | 0.72 | 42.3 | 7.12 |
| 51-2 | 481.8 | 59.3 | 14.3 | 293 | 5,70 | 0.69 | 38,0 | 7,56 |
| 51-3 | 484.4 | 45.1 | 17.6 | 331 | 4.92 | 0.71 | 37.0 | 7,25 |
| S M | 499.3 | 30.4 | 29.1 | 520 | 6.50 | 0.72 | 39,8 | 7.26 |
| 51-5 | 498.6 | 40.8 | 21.8 | 396 | 5.67 | 0.72 | 38.1 | 7.39 |
| 51-6 | 493.6 | 37.6 | 24.3 | 415 | 5.41 | 0.79 | 38.6 | 7.66 |
| 51-7 | 494.5 | 26.5 | 33.0 | 532 | 5.46 | 0.63 | 35.9 | 7.04 |
| 51-8 | 493.7 | 35.7 | 24.0 | 424 | 6.31 | 0.69 | 39.3 | 7.38 |
| 51-9 | 494.9 | 35.1 | 25.3 | 449 | 6.07 | 0.74 | 38.9 | 7.44 |
| 51-10 | 493.6 | 35.3 | 25.8 | 456 | 5.67 | 0.72 | 38.6 | 7.51 |
| 3I-11 | 500.9 | 39.6 | 24.0 | 448 | 7.01 | 0.69 | 37.4 | 7.40 |
| 31-12 | 496.7 | 31.3 | 29.7 | 486 | 6.11 | 0-67 | 39.0 | 7.04 |
| 51-13 | 497.2 | 40.8 | 23.2 | 407 | 6.95 | 0.71 | 42.1 | 7.44 |
| 51-14 | 497.9 | 65.0 | 13.3 | 280 | 7.77 | 0.66 | 32-7 | 7.23 |
| 31-15 | 490.4 | 75.8 | 11.9 | 235 | 3.54 | 0.86 | 39.7 | 8.67 |
| 51-16 | 478.7 | 37.5 | 22.3 | 390 | 4.40 | 0.82 | 41.0 | 7.57 |
| 51-17 | 491.1 | 36.4 | 23.2 | 443 | 5.65 | 0.67 | 34,8 | 7.34 |
| 51-18 | 494.3 | 41.5 | 22.9 | 420 | 6.45 | 0.76 | 41.7 | 8.01 |
| 51-19 | 498.1 | 26.9 | 33.1 | 535 | 5.32 | 0.73 | 36.9 | 7.34 |
| M-1 | 481.0 | 28.7 | 29.6 | 543 | 6.48 | 0.73 | 44,1 | 7,34 |
| M-2 | 488.2 | 51.0 | 16.8 | 365 | 6.40 | 0.70 | 38.8 | 7.51 |
| M-3 | 487.0 | 31.3 | 27.0 | 497 | 5.52 | 0.80 | 39 8 | 7,66 |
| M-4 | 491.3 | 51.2 | 18.6 | 373 | 6.63 | 0.66 | 36.3 | 7.73 |
| M-5 | 485.6 | 40.1 | 23.0 | 424 | 5.79 | 0.71 | 39.4 | 8.00 |
| M-6 | 482.0 | 41.1 | 22.8 | 433 | 6.70 | 0,63 | 36.0 | 7.73 |
| M-7 | 488.6 | 75.5 | 11.3 | 245 | 3.79 | 0.78 | 36.0 | 8.69 |
| M-8 | 489.7 | 34.6 | 25.2 | 476 | 6-72 | 0.70 | 35.4 | 7.79 |
| M-9 | 488-9 | 34.8 | 25.0 | 491 | 6.47 | 0.70 | 38.0 | 7.81 |
| **m-io** | 490.7 | 48,7 | 20.0 | 361 | 6.33 | 0.78 | 40.5 | 7.88 |
| M-11 | 482.6 | 35.0 | 24.1 | 462 | 6.52 | 0.68 | 38.5 | 738 |
| M-12 | 492.9 | 50.9 | 18.2 | 358 | 5.12 | 0,77 | 42.5 | 7.94 |
| M-13 | 487.7 | 45.3 | 19.8 | 393 | 6.88 | 0.70 | 37.1 | 7.71 |
| M-14 | 487.7 | 40.5 | 22,8 | 430 | 6.73 | 0,68 | 38,6 | 7,78 |
| M-15 | 484.2 | 77.0 | 11.5 | 246 | 3.03 | 0.83 | 38.5 | 8.69 |
| lV2-1 | 519.1 | 21.6 | 35.2 | 574 | 4.93 | 0.92 | 57.6 | 7.09 |
| lV2-2 | 515.6 | 56.5 | 15.8 | 312 | 5.66 | 0.68 | 40.8 | 7.53 |
| *2-3 | 516.7 | 54.9 | 16.8 | 333 | 5.47 | 0.68 | 45.4 | 7.26 |
| A2-4 | 513.0 | 55.1 | 16.8 | 338 | 5.72 | 0.69 | 47.6 | 7.21 |
| H2-5 | 509.1 | 37.3 | 24,7 | 453 | 6.31 | 0 78 | 39.8 | 7.61 |
| \2-6 | 515.9 | 52.1 | 17.5 | 367 | 6,75 | 068 | 37.2 | 7.60 |
| H2-7 | 515.0 | 53.7 | 18.0 | 358 | 6.34 | 0.70 | 37.7 | 7.71 |
| H2-B | 518-3 | 36.2 | 25.7 | 473 | 7.16 | 0.68 | 36,2 | 7.52 |
| *2-9 | 512.4 | 51.2 | 19.4 | 368 | 6.69 | 0.66 | 35.6 | 7.74 |
| A2-10 | 515.0 | 44.2 | 20-7 | 386 | 5.27 | 0.78 | 39.7 | 7.58 |
| A2-11 | 519.8 | 40,2 | 23-5 | 419 | 6.35 | 0,77 | 40.7 | 7-68 |
| H2-12 | 516.7 | 41.6 | 23.4 | 432 | 7.01 | 0.66 | 38.9 | 7.61 |
| A2-13 | 511.2 | 41.4 | 22.3 | 416 | 6.26 | 0.67 | 38.2 | 7.49 |
| A2-14 | 511.2 | 45.9 | 21.4 | 406 | 6.57 | 0.73 | 37.4 | 7.60 |
| *2-15 | 509.6 | 91.4 | 10.6 | 224 | 2.61 | 0.78 | 36.5 | 8.79 |
| t\2-16 | 512.2 | 38.2 | 24.5 | 456 | 6.79 | 0.70 | 39.1 | 7.58 |
| «-17 | 514.0 | 53.9 | 17.7 | 343 | 5.45 | 0.74 | 37.4 | 7.80 |
| =1-1 | 490.3 | 30.1 | 25.1 | 450 | 5.10 | 0.77 | 38.2 | 7.92 |
| =1-2 | 481.5 | 62.7 | 13.6 | 286 | 3.06 | 0.79 | 39,5 | 8.39 |
| 1-3 | 488.0 | 37.0 | 24.4 | 467 | 6.80 | 0.70 | 36.0 | 7,68 |
| 3-4 | 490.2 | 83.7 | 11.8 | 257 | 2.88 | 0-80 | 38.3 | 9.13 |



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